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Structure Refinement and Bauschinger Effect in fcc and hcp Metals

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Abstract: Although the Bauschinger effect has been investigated in some detail in various materials, the number of articles on the effect of grain size is extremely limited, and in current nanostructured materials it is practically absent. Since such materials are considered as promising for structural applications, it is important to understand their mechanical behavior under conditions of changing the direction of deformation, and, therefore, it is necessary to study the Bauschinger effect and its dependence on grain size. The Bauschinger effect was investigated by a single exemplary method for tensile compression of commercially pure hcp titanium and fcc copper, with different grain sizes in the range from hundreds of microns to hundreds of nanometers. The change in grain size was performed by structure refinement by the method of severe plastic deformation using equal-channel angular pressing and subsequent annealing. It has been established that, in both materials, the Bauschinger effect increases with a decrease in grain size, the degree of permanent strain and the duration of exposure between forward and reverse deformation. The signs of the Bauschinger parameter in copper and titanium are opposite. The relationship between the Bauschinger effect and the nature of strain hardening in titanium and softening in copper in the ultrafine-grained state is discussed.

Keywords: titanium; copper; microstructure; grain size; tension; compression; yield stress

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1. Introduction

Alternating deformation of metallic materials leads to the phenomenon of fatigue, which is characterized by a decrease in the service life or duration of operation of the structural element before destruction. In this case, one of the ways to quantify the sensitivity of the material to the manifestation of fatigue is the magnitude of the Bauschinger effect [1,2], which shows a decrease in deformation resistance when changing the direction of deformation, for example, tension–compression. Experimental studies show that the absolute difference in the yield range of materials with the Bauschinger effect under tension and compression in opposite directions can reach 25–30% [3].

It is assumed that the Bauschinger effect reflects the combined effect of the deformation processes of hardening and softening, and that it is caused by structure features and internal stresses [2]. In this regard, the study of factors influencing cyclic behavior is of great importance for the theory of plasticity, as well as for determining the stress state in the practice of metal forming. As a result of tension and unloading from a certain level of flow stresses, and then changing the direction of deformation to compression, the yield stress becomes less than in tension. The Bauschinger effect is considered ideal to be zero if the decrease in compressive yield stress is equal to the increase in tensile yield stress.

As shown by numerous original articles and reviews [4–11], the Bauschinger effect depends on the material itself [5,6], its properties, microstructure (grain size) [7,8], type of crystal lattice [9], deformation conditions (type of stress–strain state), heat treatment [10], speed, degree and direction of permanent strain [11]. It should be noted that, for most of these factors, their specific effect on the Bauschinger effect remains debatable and often opposite. This applies primarily to the magnitude of the permanent strain and the grain size. For example, in [12] it was argued that, in pearlite steels, the Bauschinger effect increases with increasing degree of permanent strain, which coincides with the result in [1]. On the contrary, in [11], a decrease in the Bauschinger effect was found for steels of different chemical composition. Similar contradictions exist for the role of grain size. Thus, in [9], the author believed that the grain size in face-centered and body-centered cubic metals Ni, Cu, Al and steel does not affect the Bauschinger effect. This is the opposite of the conclusions of the work on the reduction and even disappearance of the Bauschinger effect with an increase in grain size in steel, aluminum, copper, magnesium and zinc [13]. In [14], the authors also found that the Bauschinger effect increases with decreasing grain size in copper.

In contrast to copper, studies of the Bauschinger effect in pure titanium are extremely limited and are mainly associated with the possibility of twinning or high-speed loading in titanium alloys [15–17]. Direct work on the study of the grain size effect on the Bauschinger effect in pure titanium has not been found. It should be noted here that, in the above works, the size effect was investigated for materials in a coarse-grained state, in which the grain size was tens and even hundreds of microns.

With the development of a new direction for the production of materials with an ultrafine-grained structure, the importance of such studies has become even higher, since the strength of materials can be multiplied by reducing the grain size in the nanosized region. One of the first works in this direction was an experimental study [18], in which the authors measured the energy parameter of the Bauschinger effect under fatigue loading using the example of massive ultrafine-grained copper. It has been shown that the Bauschinger effect increases with decreasing grain size and depends on the degree of nonequilibrium of the grain boundaries. In a recent paper [19], a study of the temperature dependence of the Bauschinger effect in the range of 300–600 K was carried out by molecular dynamics in a nano-single crystal aluminum. It has been shown that the Bauschinger effect weakens significantly with increasing temperature.

The closest to the present work was the study of the dependence of the Bauschinger effect on grain size in ultrafine-grained aluminum obtained by equal-channel angular pressing [20]. The authors also confirmed an increase in the Bauschinger parameter with a decrease in grain size in the range from 6 to 0.56 μm. As for the mechanism of the Bauschinger effect, there are several main hypotheses in the literature described in the review [4]. Common to the hypotheses put forward is the formation of internal stresses of different levels of long-range action and the dislocation movement at different scale levels. The earliest hypothesis is the idea of residual-oriented macrostresses in the volume of a solid state due to inhomogeneity of primary deformation [21]. Another hypothesis is related to the formation of microstresses caused by dislocation clusters, not only in polycrystals, but also in single crystals [22]. Recently, a hypothesis has appeared about the residual deformation of the crystal lattice on the scale of the grain group, caused by the anisotropy of elastic and plastic deformation [7].

It is necessary to note the appearance of a number of articles on the effect of gradient microstructure, including nanostructure, on the Bauschinger effect. So, in [23], the authors report that the gradient-structured copper exhibits an extraordinarily large Bauschinger effect in comparison with homogeneous structure. The large Bauschinger effect was also observed by the authors of [24] in in situ TEM straining experiments on freestanding gold and aluminum films to explore the role of microstructural heterogeneity in the deformation behavior of nanocrystalline metals. In both papers, the preliminary deformation exceeded a multiple of 0.2%, which is nontypical for a classical Bauschinger effect.

One of the new trends in materials science is the study of high-entropy single-phase alloys. In [25], the authors study the contribution of kinematic hardening based on the Bauschinger effect and attribute it to the low probability of cross-slip in such materials. A similar approach is used in modeling the Bauschinger effect in [26,27].

The purpose of this article is to investigate the role of structure refinement and test conditions for inelastic tensile–compressive behavior in metals with different types of crystal lattice.

2. Materials and Methods

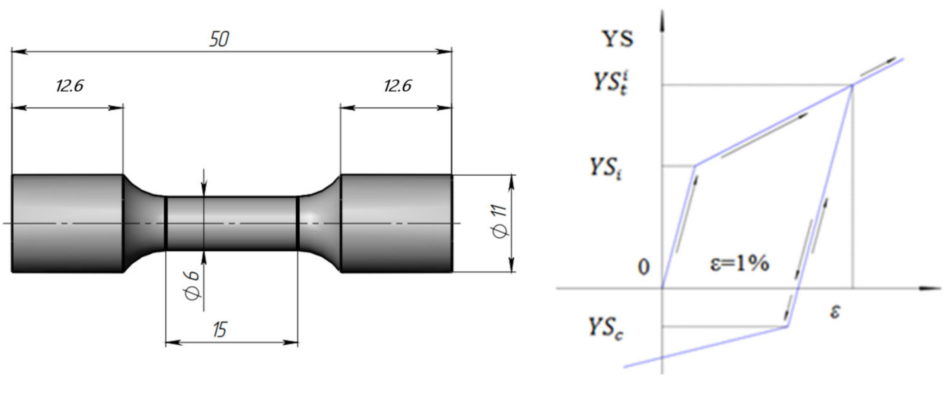
The materials of the study were hot-rolled rods with a diameter of 40 mm made of commercially pure titanium (99.5%) and copper M1 (99.9%), which have markedly different strength characteristics and the type of crystal lattice, respectively, hcp and bcc. The chemical composition of both metals is presented in Table 1. The cross-sectional grain size of the rods in the delivery state was 10 and 20 µm, respectively, for titanium and copper.

Table 1. Chemical composition of titanium and copper (wt %) in an as-received state.

Materials	Fe	O	N	Si	C
titanium	0.18	0.12	0.04	0.1	0.07
copper	0.005	0.05	-	-	-

To refine the microstructure, the initial rods after mechanical turning to a diameter of 20 mm were subjected to multipass equal-channel angular pressing (ECAP) at room temperature (for copper) and 450 °C (for titanium). Details of the ECAP process can be found in [28,29]. To obtain a homogeneous coarse-grained structure, as well as to eliminate the internal stresses, the ECAP workpieces were annealed in a vacuum furnace at a temperature of 600 °C (for titanium and copper) and 300 °C (for titanium only [28]) for 1 h.

Mechanical tests in accordance with GOST 1497-84 were performed on a horizontal electromechanical machine IR 5047-50 (JSC "TOCHPRIBOR", Ivanovo, Russia) with computer control in the sequence of tension–compression–tension at room temperature and strain rate of 1×10^{-3} s^{-1} on the same sample cut in the longitudinal direction of the bar. A similar test technique was used in [30]. Cylindrical specimens cut in the longitudinal direction of the rod were used, the dimensions of the working part of which were 6 mm in diameter and 15 mm in length (Figure 1a). A strain gauge was placed on the working part of the sample, which made it possible to measure axial deformation with an accuracy of ±0.005%.



(**a**) (**b**)

Figure 1. Sample view and dimensions for tension–compression (**a**) and loading scheme (**b**).

A diagram of the sequence of loading stages is shown in Figure 1b. The first loading of the specimen was performed by tension to permanent deformation ε = 1.0%. After fixing the corresponding flow stress YS_t, unloading and holding for τ = 60 min, a second loading was performed in the opposite direction by compression to flow stress YS_c. The third loading was performed by re-tension almost without stopping. Additionally, in the ECAP state, parameters τ = 1 min and ε = 0.2% were used. The Bauschinger effect was estimated by two different dimensionless parameters:

$$\beta_1 = YS_c/YS_t^1 \quad (1)$$

$$\beta_2 = YS_t^1 - YS_t^2/YS_t^1, \quad (2)$$

YS_t^1—yield stress at the first loading by tension; YS_c—yield stress at the compression; YS_t^2—yield stress at the second tension.

The number of test samples was two for each structural state and loading mode, and the accuracy of determining the parameters β_1 and β_2 was ±0.02. The microstructure was investigated by optical metallography (NEOPHOT-2 microscope, Carl Zeiss, Oberkochen, Germany) and transmission electron microscopy (TEM) of foils in the transverse direction (JEM 100B microscope, JEOL Ltd., Tokyo, Japan). Electron diffraction patterns were taken from an area of 2 μm^2. The average grain size was measured by number-averaging the diameters of more than 60 grains. The titanium samples for light microscopy were etched in an electrolyte consisting of 100 mL of H_2O, 2 mL of HF and 5 mL of H_2O_2. For TEM of titanium foils, double-sided jet electropolishing in a solution of 5% perchloric acid, 35% butanol and 60% methanol at a temperature of −30 °C was applied.

Copper samples for TEM were thinned by mechanically grinding and argon ion-beam in a solution of 25% phosphoric acid, 25% ethanol and 50% water. Liquid nitrogen was used to prevent any heating of the TEM specimens during the ion-beam thinning process. Samples out of copper for optical microscopy were prepared by grinding, polishing and etching in the nitric acid solution.

3. Results

3.1. Microstructure

Figure 2 presents the results of a study of the microstructure features and the average grain size of titanium and copper in an as-received state and the ultrafine-grained state obtained by the ECAP.

An as-received state is characterized by a partially recrystallized structure with an average grain size of 25 µm in titanium and 30 µm in copper, as well as the presence of twins particularly numerous in titanium (Figure 2a,b). The microstructure in the ultrafine-grained state in both materials (Figure 2c,d) is heterogeneous, predominantly fragmented, an increased density of dislocations is observed in the grains, twins are practically not observed, and the average grain (subgrain) size is approximately the same and is 300 nm. The circular nature of the arrangement of electronic reflexes in microdiffraction patterns confirms the strong structure refinement.

Figure 3 shows the microstructures of copper and titanium subjected to equal-channel angular pressing in the state after annealing at 600 and 300 °C for 1 h. Post-deformation annealing at 600 °C results in a fully recrystallized structure with straight grain boundaries and an average grain size of 50 and 100 µm, respectively, for titanium and copper (Figure 3a,b).

The observed annealing twins are typical structural elements in both metals. Their shape in both metals is different: thin in titanium and wide in copper. Post-deformation annealing in ultrafine-grained titanium at a lower temperature of 300 °C for 1 h did not affect the average grain size, the grain boundaries became clearer, but the density of dislocations within the grains decreased (Figure 3c).

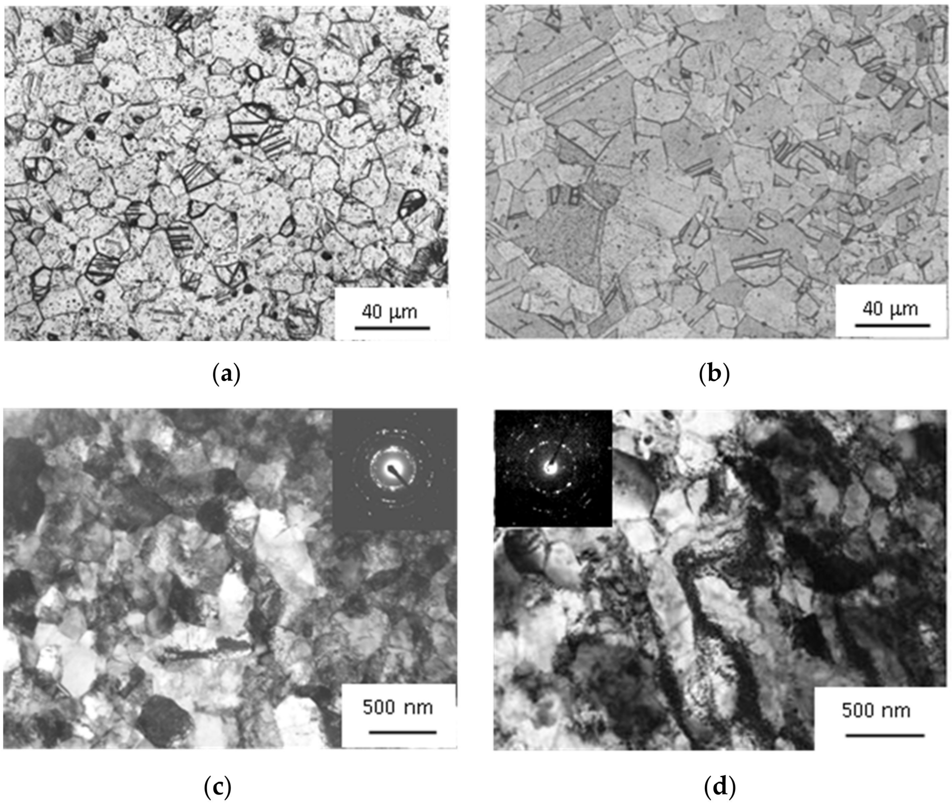


Figure 2. Microstructure and microdiffraction images in cross section for titanium (**a**,**c**) and copper (**b**,**d**) in states: (**a**,**b**)—as-received (optical); (**c**,**d**)—ultrafine-grained (TEM).

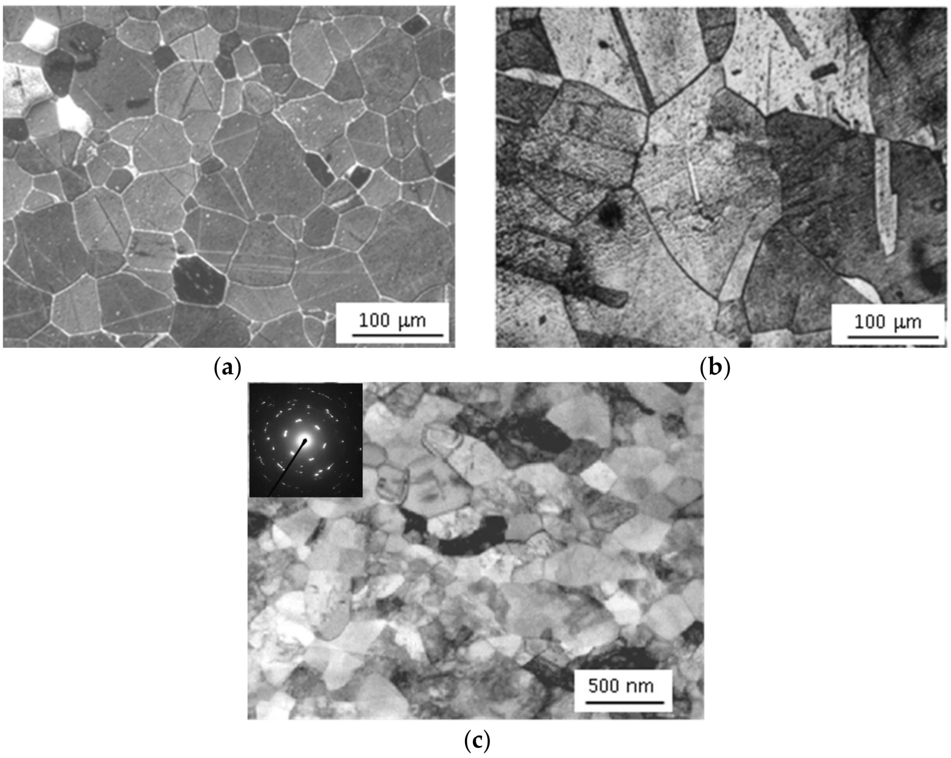
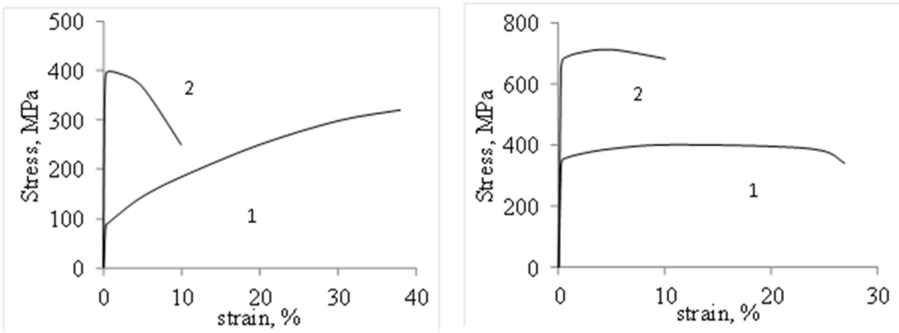


Figure 3. Microstructure images in cross section for ECAP titanium (**a**,**c**) and ECAP copper (**b**) in states: (**a**,**b**)—after annealing at 600 °C (optical); (**c**)—after annealing at 300 °C (TEM).

3.2. Tension Tests

Figure 4 shows the tensile stress–strain curves of titanium and copper samples in the as-received state and the ultrafine-grained state obtained by equal-channel angular pressing.



(**a**) (**b**)

Figure 4. Typical stress–strain curves for copper (**a**) and titanium (**b**) in as-received state (curve 1) and ultrafine-grained state (curve 2).

It can be seen that the microstructure refinement of both materials led to a strong change in mechanical properties: a multiple increase in strength/yield stress and a multiple decrease in elongation to failure. In addition, the shape of the tensile curve has changed in copper. Instead of an extended strain hardening and uniform deformation stages, a short softening stage and neck formation (localized deformation) appeared in the ultrafine-grained state. Unlike copper, the stage of strain hardening in titanium has been preserved, although it has also been greatly reduced.

3.3. Bauschinger Effect

Table 2 and Figure 5 show the results of determining the Bauschinger parameters for states with different grain sizes. For both metals in all states (except for the ECAP of titanium annealed at 600 °C), the parameter $\beta_1 < 1$ is always positive, in contrast to the β_2 parameter, and tends to decrease with decreasing grain size in the range from hundreds of microns to hundreds of nanometers. The β_2 parameter in both metals increases with decreasing grain size, i.e., compression deformation in both metals increases the difference in flow stresses during primary and repeated tension (Table 2).

Table 2. Bauschinger parameters for Ti and Cu in different structure states and test conditions.

Treatment Method	d, μm	τ, min	ε, %	Bauschinger Parameters	
				β_1	β_2
copper					
ECAP + annealing at 600 °C–1 h	100	60	1	0.87	+0.04
As-received state	30	60	1	0.80	+0.13
ECAP	0.3	60	1	0.95	+0.20
	0.3	1	0.2	0.72	+0.26
titanium					
ECAP + annealing at 600 °C–1 h	50	60	1	1.08	−0.28
As-received state	25	60	1	0.95	−0.21
ECAP	0.3	60	1	0.83	−0.07
	0.3	1	0.2	0.91	+0.05
ECAP + annealing at 300 °C–1 h	0.3	60	1	0.86	−0.11

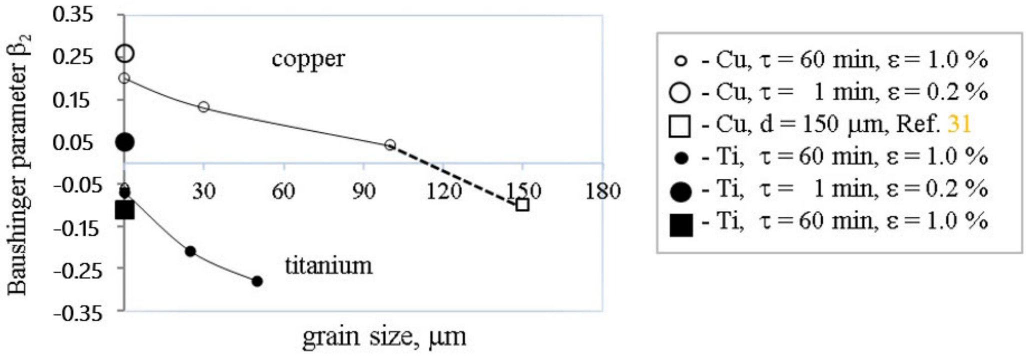


Figure 5. Grain size dependance of Bauschinger parameter β_2 in copper and titanium, data from [31].

An interesting fact is that the sign of the β_2 parameter for the studied metals is different: it is positive for copper and negative for titanium in the entire studied grain size range (Figure 5). In the same figure, based on the literature data [31], the dotted part of the curve for Cu with a grain size of 150 μm is shown. It can be seen that the literature data are in good agreement with the experimental ones and indicate the possibility of changing the sign of the parameter β_2 with an increase in the grain size. Another feature is the influence of loading conditions: the degree of permanent deformation (ε) and the holding time (τ) between the forward and reverse test. A decrease ε from 1 to 0.2% and a decrease τ from 60 to 1 min contributes to an increase in the β_2 parameter for both metals in the ultrafine-grained state and even a change of sign in titanium (Table 2).

On the contrary, annealing at 300 °C for 1 h in ultrafine-grained titanium reduces the β_2 parameter. In general, the maximum absolute value of the Bauschinger effect in the studied metals can be more than 25%.

4. Discussion

A comparison of the individual structural elements in titanium and copper, respectively for each structural state, shows their morphological identity. Thus, in an as-received state for both metals, the average grain size (25–30 μm) and the density of twins are almost close (Figure 2a,b). In the state after severe plastic deformation by the ECAP method, the grain size (0.3 μm) and the equiaxed grain shape in copper and titanium are identical, and there are no twins (Figure 2c,d). Post-deformation annealing at 600 °C led to the reduction of twins and recrystallization grain growth stronger in copper (100 μm) than in titanium (50 μm) (Figure 3a,b). It should be noted that the structural states differ not only in the grain size, but also in the density of dislocations.

The ultrafine-grained structure in contrast to the fully recrystallized structure is fragmented and contains an increased density of intragranular dislocations and low-angle grain boundaries (Figure 2c,d). Nevertheless, the microdiffraction pattern in the form of rings consisting of evenly spaced reflexes of varying intensity indicates the preferential formation of high-angle grain boundaries. Structure elements of this type introduce additional internal stresses that increase the resistance to the movement of dislocations and can affect the Bauschinger effect. In this regard, the post-deformation annealing performed at 600 °C in both metals was recrystallization and not only relieved internal stresses, but sharply increased the grain size and contributed to the appearance of annealing twins (Figure 3a,b). As for the annealing of ECAP titanium at 300 °C (Figure 3c), it contributed to the removal of stresses, a decrease in the density of dislocations without changing the grain size and the formation of microstructure in accordance with [29].

The study of the Bauschinger effect made it possible to establish a number of features that are important for understanding the nature of the phenomenon. In this case, the estimation of the Bauschinger effect was performed using two parameters, β_1 and β_2. The first, the simplest parameter, is often given in the literature as an indicator of the SD (strength-different) effect, which evaluates the anisotropy (asymmetry) of the yield stress and the Bauschinger effect. A decrease in the β_1 parameter with a decrease in grain size indicates a significant increase in the anisotropy of the yield stress in both metals. As can be seen from Table 2, the degree of anisotropy (SD effect) can reach 28% in the isotropic fcc lattice of copper and 17% in the anisotropic hcp lattice of titanium. The existing single deviations from this trend for ultrafine-grained titanium and copper have not been studied in the framework of this work and require special attention in the future.

Despite the correlation of both parameters with each other, the second parameter β_2 turned out to be more informative. It has been experimentally shown that the Bauschinger effect is enhanced by structure refinement of metals with different types of crystal lattice, most significantly in the region close to nanoscale (Figure 5). Since this fact was previously questioned or concerned only in the field of coarse-grained materials, the results obtained in this work allow us to extend the view to the nature of the Bauschinger effect for nanoscale materials. It is known from the literature that the basis of the proposed mechanism of the Bauschinger effect is formed by internal stresses and microstructural features [2]. Structure refinement to a greater or lesser extent is always accompanied by an increase in internal stresses. Therefore, the obtained results only confirm the previously put forward hypotheses regarding internal stresses.

An interesting fact was that, despite the same tendency to enhance the Bauschinger effect during the structure refinement, the sign of the parameter β_2 in the studied metals is opposite in the studied range of grain sizes (Figure 5). A similar result was presented for copper with grain sizes of 60 and 150 μm in [31], where, however, the sign of the Bauschinger effect changed from negative to positive with increasing grain size. Analysis of the experimental conditions showed that the author used the "compression-tension-compression" scheme in contrast to the "tension-compression-tension" scheme in this article. In our case, regardless of the grain size, a change in the direction of deformation in copper leads to softening, and in titanium to hardening.

The grain size, at which the parameter β_2 approaches zero, can be considered as critical, at which point there is a change in the sign of the parameter, and, consequently, the contribution of the predominant deformation mechanism, hardening or softening. One of the possible explanations for the results obtained may be a different type of crystal lattice, which in titanium is an anisotropic hcp, and in copper an isotropic fcc. As a consequence, the number of sliding systems in titanium is extremely limited compared to copper. This difference is especially pronounced in the crystallographic texture of copper and titanium as a result of severe plastic deformation by the ECAP method. As is known, in the ECAP titanium, it is associated with basic {0001} and pyramidal {1012} sliding systems, and in the ECAP copper with <110> {111} sliding systems [32]. A special role in titanium can be played by the mechanism of twinning in the ECAP state, which is activated during compression. It is known that the shear stress during twinning is markedly higher than during sliding.

Note the importance of test conditions in assessing the Bauschinger effect in the ultrafine-grained state of materials (Table 2). A decrease in permanent deformation and holding time between tension and compression led to an increase in the β_2 parameter in both metals, which is due to additional internal stresses and is consistent with the literature data for conventional polycrystalline metals. Removal of internal stresses as a result of annealing at 300 °C in ultrafine-grained titanium with a constant grain size leads to the opposite effect of reducing the Bauschinger effect.

To understand the reason for the difference in the manifestation of the Bauschinger effect in copper and titanium, the type of tensile curves in Figure 4, which differ in the nature of strain hardening, especially in the ultrafine-grained state, it can be seen that in copper, strong strain hardening in the coarse-grained as-received state is replaced by strong softening in the ultrafine-grained state almost immediately after reaching the yield stress. On the contrary, in titanium, the nature of strain hardening with the structure refinement remains the same.

Comparing the results obtained with the study for ultrafine-grained aluminum in [21], it can be noted that the enhancement of the Bauschinger effect with a decrease in grain size in the present work is confirmed even in a wider range of grains. However, for the ultrafine-grained state, the effect of permanent deformation in the range from 0.2 to 1% on the Bauschinger effect in copper and titanium turned out to be opposite to the effect in aluminum (Table 2). A possible reason for the discrepancy in the results could probably be the different degree of nonequilibrium of the grain boundaries in the annealed state for aluminum and the unannealed state for copper and titanium.

Understanding and adjusting the sign of the Bauschinger effect by forming the necessary microstructure and deformation conditions can be useful for the design of metal forming processes or increasing resistance under cyclic loading. The different sign of the Bauschinger parameter β_2 in copper and titanium is in good agreement with their fundamentally different multicycle fatigue behavior in the ultrafine-grained state, which in copper is due to wave slip along many planes, and in titanium by sliding along prismatic planes [33].

5. Conclusions

For the first time, the effect of structural refinement and test conditions on the Bauschinger effect at small plastic deformations in the tension–compression in pure copper and titanium with different types of crystal lattice has been studied in detail. The following conclusions can be drawn:

1. It is confirmed that in commercially pure polycrystalline metals titanium and copper, a decrease in the average grain size in the range from tens of microns to hundreds of nanometers leads to an increase in the Bauschinger parameter β_2 (an increase in the difference in flow stresses in one direction after deformation in the opposite direction).
2. The opposite sign of the Bauschinger effect in copper and titanium in a wide grain-size range indicates the predominant deformation mechanism acting when the direction of deformation changes. In copper, such a mechanism is the most common typical softening, whereas in titanium, anomalous hardening is observed. A decrease in the magnitude of permanent deformation and the duration of exposure between direct and reverse loading contributes to an increase in the Bauschinger parameter in both metals and a change from a negative sign to a positive one.
3. It is assumed that the different sign and magnitude of the Bauschinger effect in ultrafine-grained copper and titanium are associated with a different type of crystal lattice and, as a result, a type of crystallographic texture.

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Data Availability Statement: Data are available in a publicly accessible repository.

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References

1. Bauschinger, J. Über die Veränderung der Elastizitätsgrenze und des Elastizitätsmoduls verschiedener Metalle. In *Civilingenieur XXVII*; Felix: Leipzig, Germany, 1881; pp. 289–348.
2. Tan, Z.; Magnusson, C.; Persson, B. The Bauschinger effect in compression-tension of sheet metals. *Mater. Sci. Eng. A* **1994**, *183*, 31–38. [CrossRef]

3. Kulawinski, D.; Nagel, K.; Henkel, S.; Hubner, P.; Kuna, M.; Biermann, H. Characterization of stress-strain behavior of a cast TRIP steel under different biaxial planar load rations. *Eng. Fracture Mech.* **2011**, *78*, 1684–1695. [CrossRef]
4. Adigamov, R.R.; Andreev, V.A.; Rogachev, S.O.; Fedotov, E.S.; Khadeev, G.E.; Yusupov, V.S. Bauschinger effect during alternating deformation. *Izv. Ferr. Met.* **2022**, *65*, 455–466. (In Russian) [CrossRef]
5. Moan, G.D.; Embury, J.D. A study of the bauschinger effect in Al–Cu alloys. *Acta Met.* **1979**, *27*, 903–914. [CrossRef]
6. Grachev, S.V.; Kazyaeva, I.D.; Pumpyanskii, D.A. Thermal stability of the Bauschinger effect in the cold-deformed steel. *Phys. Met. Metallogr.* **2004**, *97*, 217–219.
7. Mamun, A.; Moat, R.; Kelleher, J.; Bouchard, P. Origin of the Bauschinger effect in a polycrystalline material. *Mater. Sci. Eng. A* **2017**, *707*, 576–584. [CrossRef]
8. Trelewicz, J.R.; Schuh, C.A. The Hall–Petch breakdown in nanocrystalline metals: A crossover to glass-like deformation. *Acta Mater.* **2007**, *55*, 5948–5958. [CrossRef]
9. Woolley, R.L. The Bauschinger effect in some face-centered and body-centered cubic metals. *Philos. Mag.* **1953**, *44*, 597–618. [CrossRef]
10. Skelton, R.P. Bauschinger yield in the range 400–1025 °C during cyclic deformation of high temperature alloys. *Mater. High Temp.* **2013**, *30*, 241–260. [CrossRef]
11. Kim, H.L.; Park, S.H. Loading Direction Dependence of Yield-Point Phenomenon and Bauschinger Effect in API X70 Steel Sheet. *Met. Mater. Int.* **2020**, *26*, 14–24. [CrossRef]
12. Toribio, J.; Kharin, V.; Ayaso, F.-J.; Lorenzo, M.; González, B.; Matos, J.-C.; Aguado, L. Analysis of the Bauschinger Effect in Cold Drawn Pearlitic Steels. *Metals* **2020**, *10*, 114. [CrossRef]
13. Ratner, S.I.; Danilov, Y.S. Changes of proportional limits and yield point at repeated loading. *Zavod. Lab.* **1950**, *4*, 46–47. (In Russian)
14. Mahato, J.K.; De, P.S.; Sarkar, A.; Kundu, A.; Chakraborti, P.C. Effect of deformation mode and grain size on Bauschinger behavior of annealed copper. *Int. J. Fatigue* **2016**, *83*, 42–52. [CrossRef]
15. Canel, G.I.; Savinykh, A.S.; Garkushin, G.V.; Razorenov, S.V. High-speed deformation of titanium in shock waves at normal and elevated temperatures. *ZhETF* **2021**, *159*, 524–532. [CrossRef]
16. Sole, N.C. An Investigation on the Bauschinger Effect in Titanium Alloys for Aerospace Applications: Strain Rate and Pressure Dependence. Master's Thesis, University of Oxford, Oxford, UK, 2021; 124p.
17. Yu, X.; Ji, H.; Zhang, H.; Xu, D. Experimental and numerical investigation on the Bauschinger effect during cold forming of TC4 ELI alloy. *Int. J. Mater. Form.* **2022**, *15*, 16. [CrossRef]
18. Vinogradov, A.; Kaneko, Y.; Kitagawa, K.; Hashimoto, S.; Stolyarov, V.; Valiev, R. Cyclic response of ultrafine-grained copper at constant plastic strain amplitude. *Scr. Mater.* **1997**, *36*, 1345–1351. [CrossRef]
19. Shen, J.; Zhou, J.; Zhao, G.; Gong, C.; Yu, J.; Xia, X.; Xian, F. Attenuation of the Bauschinger effect and enhancement of tension–compression asymmetry in single crystal aluminum by temperature. *RSC Adv.* **2022**, *12*, 21235–21246. [CrossRef]
20. Gao, S.; Yoshino, K.; Terada, D.; Kaneko, Y.; Tsuji, N. Significant Bauschinger effect and back stress strengthening in an ultrafine grained pure aluminum fabricated by severe plastic deformation process. *Scr. Mater.* **2022**, *211*, 114503. [CrossRef]
21. Brown, L.M. Orowan's explanation of the Bauschinger effect. *Scr. Met.* **1977**, *11*, 127–131. [CrossRef]
22. Hu, X.; Way, C.; Margolin, H.; Nourbakhsh, S. The Bauschinger effect and the stresses in a strained single crystal. *Scr. Met. Mater.* **1992**, *27*, 865–870. [CrossRef]
23. Liu, X.; Yuan, F.; Zhu, Y.; Wu, X. Extraordinary Bauschinger effect in gradient structured copper. *Scr. Mater.* **2018**, *150*, 57–60. [CrossRef]
24. Rajagopalan, J.; Rentenberger, C.; Karnthaler, H.P.; Dehm, G.; Saif, M.T.A. In situ TEM study of microplasticity and Bauschinger effect in nanocrystalline metals. *Acta Mater.* **2010**, *58*, 4772–4782. [CrossRef]
25. Bouaziz, O.; Moon, J.; Kim, H.S.; Estrin, Y. Isotropic and kinematic hardening of a high entropy alloy. *Scr. Mater.* **2020**, *191*, 107–110. [CrossRef]
26. Bouaziz, O.; Kim, H.S.; Lee, J.; Estrin, Y. Bauschinger Effect or Kinematic Hardening: Bridging Microstructure and Continuum Mechanics. *Met. Mater. Int.* **2022**, *29*, 280–292. [CrossRef]
27. Roostaei, A.A.; Jahed, H. Experimental observations in cyclic loading of metals. In *Cyclic Plasticity of Metals*; Jahed, H., Roostaei, A.A., Eds.; Elsevier Series on Plasticity of Materials; Elsevier: Amsterdam, The Netherlands, 2022; pp. 3–22. [CrossRef]
28. Stolyarov, V.V.; Zhu, Y.; Alexandrov, I.V.; Lowe, T.C.; Valiev, R.Z. Influence of ECAP routes on the microstructure and properties of pure Ti. *Mater. Sci. Eng. A* **2001**, *299*, 59–67. [CrossRef]
29. Valiev, R.; Kozlov, E.; Ivanov, Y.; Lian, J.; Nazarov, A.; Baudelet, B. Deformation behaviour of ultra-fine-grained copper. *Acta Met. Mater.* **1994**, *42*, 2467–2475. [CrossRef]
30. Khvan, A.D.; Khvan, D.V.; Voropaev, A.A. One-sample method of evaluation of Bauschinger effect. *Ind. Lab. Diagn. Mater.* **2020**, *86*, 55–58. [CrossRef]
31. Danilov, Y.S. Influence of grain size on Bauschinger effect. *Met. Sci. Heat Treat.* **1964**, *6*, 563–565. [CrossRef]

32. Alexandrov, I.V.; Sitdikov, V.D. Crystallographic Texture Development in CP Ti Subjected to ECAP. *Mater. Sci. Forum* **2008**, *584–586*, 765–770.
33. Vinogradov, A.Y.; Stolyarov, V.V.; Hashimoto, S.; Valiev, R.Z. Cyclic behavior of ultrafine-grain titanium produced by severe plastic deformation. *Mater. Sci. Eng. A* **2001**, *318*, 163–173. [CrossRef]